Grain Size of Metallic Strip

1. SCOPE

1.1. Content

This specification covers the test procedure for determining the grain size of metal strip.

1.2. Reference Documents

- 100-746: Material Specification (Phosphor Bronze Strip, UNS C51000, Special Grain Size)
- ASTM E112: Standard Test Methods for Determining Average Grain Size
- ISO 2624: Copper and Copper Alloys - Estimation of Average Grain Size, Second Edition

1.3. Descriptions

- 109-89-1: Estimating Grain Size visually shall be used as the primary method of determining grain size for metallic strip.
- 109-89-2: Linear Intercept (Heyn), unless otherwise specified in the referencing document, this procedure shall be used as a referee method only.
- 109-89-3: Linear Intercept (Abrams), unless otherwise specified in the referencing document, this procedure shall be used as a referee method only.

2. TEST EQUIPMENT

Metallograph: Capable of magnification of 75 to 1600X

3. TEST SPECIMEN

Specimen to be examined shall have the surface polished and etched using any technique that will result in clearly distinguishable grain boundaries. Surface to be examined is indicated in Figure 1.

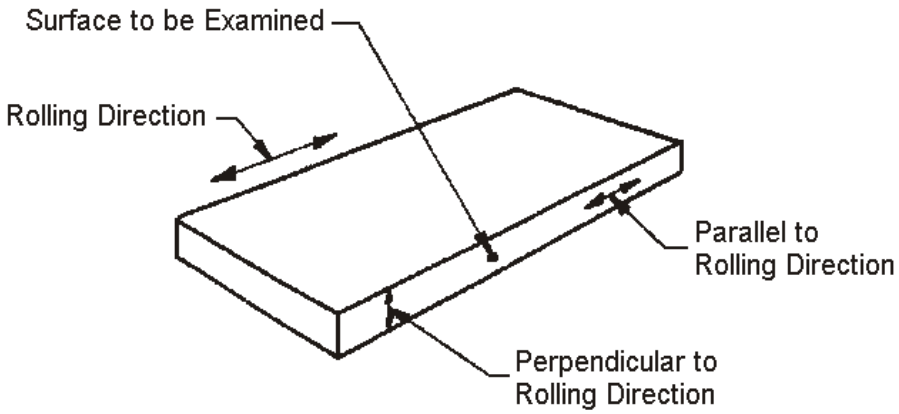

Figure 1

4. TEST PROCEDURE

The characterization and understanding of the properties of a material involve an unbiased and accurate assessment of the structure of the material. The importance of an accurate grain size determination and the influence of grain size on the material properties are evidenced by the common use of the Hall-Petch relationship between strength and grain size. Accordingly, it would be extremely valuable to have a fast and accurate method of measuring the grain size of a material. To be of practical value, such a method should require a minimum number of observations, provide data having statistical significance, and be applicable to a wide range of single-phase microstructures. These requirements are met by the procedure described and demonstrated in this specification.

4.1. Visual Method, 109-89-1

A. Prepared specimen shall be compared visually to Figure 12 for nonferrous materials and Figure 13 for ferrous materials.

B. In the event of conflict, either method 109-89-2 or 109-89-3 shall be used as the referee method.

4.2. Heyn's Intercept Method, 109-89-2

A. Prepared specimen surface shall be examined under magnification using a metallograph. Select a magnification that will result in an intercept count within the range shown in Figure 3.

B. Viewed area shall either be photographed or displayed on a viewing screen at the selected magnification.

1. Photograph shall have 2 thin lines, 100 millimeters long, placed on it. One line shall be parallel to the rolling direction and the other perpendicular as indicated in Figure 2.

2. Viewing screen shall be capable of having a thin, 100 millimeters long, line superimposed on the image parallel to the rolling direction and perpendicular as indicated in Figure 2.

C. Count the number of grains which intercept the line in each direction and record. Grains touched by the ends of a line count as half grains. If the number of grains intercepted by the line is outside the range of intercepts shown in Figure 3, increase or decrease magnification until the intercepts are within the range.

D. Grain size measurements shall be made on 2 representative fields from each specimen. If the specimens have a duplex grain size, measurements shall be made in each representative area.

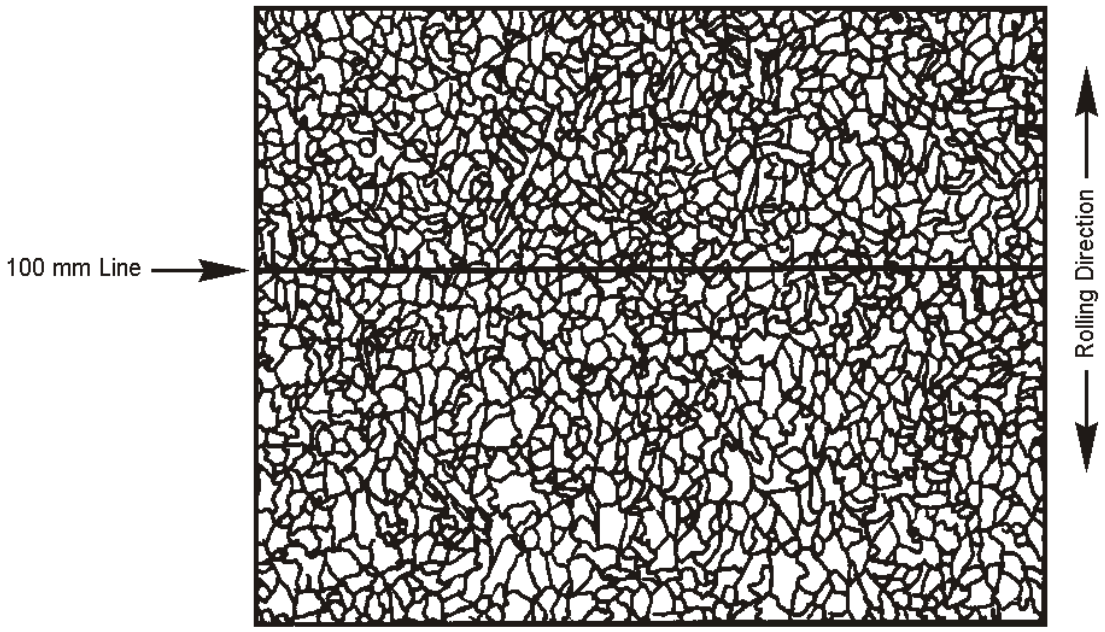

Figure 2
1600X Magnification

E. Determine grain size in millimeters as follows:

1. Find the closest number of intercepts, for each direction, from Figure 3 and record the grain direction. Figure 3 is at 100X magnification. Use Method 2 if other than 100X magnification is used.

Intercepts	Grain Diameter (millimeters)	Intercepts	Grain Diameter (millimeters)	Intercepts	Grain Diameter (millimeters)
12.5	.090	37.6	.030	125	.0090
14.9	.075	42.0	.027	141	.0080
16.1	.070	45.1	.025	161	.0070
17.1	.065	50.0	.022	168	.0067
18.8	.060	56.4	.020	188	.0060
21.0	.055	59.5	.019	200	.0056
22.6	.050	70.7	.016	226	.0050
25.0	.045	75.2	.015	238	.0047
28.2	.040	84.1	.013	283	.0040
29.7	.038	100	.011	336	.0033
32.2	.035	113	.010	376	.0030
35.4	.032	119	.0094	400	.0028
				451	.0025

Figure 3
At 100X Magnification

2. Calculate the grain size for each direction as follows:

$$Grain\ Size\ (mm) = [Grain\ Diameter\ (mm)]\ X\ \frac{100X}{Selected\ Magnification}$$

3. Calculate average grain size by adding the grain sizes parallel and perpendicular to rolling direction and divide by 2 as follows:

$$Average\ Grain\ Size\ (mm) = \frac{\parallel\ Direction\ Grain\ Size\ +\ \perp\ Direction\ Grain\ Size}{2}$$

4. Rounding Off Procedure

The combined grain size shall be expressed to the nearest .0001 millimeter. In the reporting procedure, round off to the nearest .001 millimeter grain size.

Example: .0044 millimeters rounds off to .004 millimeters
.0045 millimeters rounds off to .005 millimeters

F. Example

The following example is for 37 grain intercepts in the direction perpendicular to the rolling direction and 25 grain intercepts parallel to the rolling direction. The intercept count was made at 1600X magnification.

1. Perpendicular Direction Grain Size

From Figure 3, for 37 grains, the closest grain diameter is .030 millimeters. Using the equation in paragraph 4.2.E.2., determine the grain size as follows:

$$Grain\ Size\ \perp\ Direction = (.030)\ X\ \left(\frac{100X}{1600X}\right) = .0019mm$$

2. Parallel Direction Grain Size

From Figure 3, for 25 grains, the closest grain diameter is .045 millimeters. Using the equation in paragraph 4.2.E.2., determine the grain size as follows:

$$Grain\ Size\ \parallel\ Direction = (.045)\ X\ \left(\frac{100X}{1600X}\right) = .0028mm$$

3. Average Grain Size

Using the equation in paragraph 4.2.E.3., determine the average grain size as follows:

$$Average\ Grain\ Size = \frac{.0019\ +\ .0028}{2} = .0023mm$$

4.3. Abrams Intercept Method, 109-89-3

A. The procedure is based on the intercept method and utilizes a 50 cm total length circular pattern consisting of three concentric circles whose radii obey an arithmetic progression. The statistical advantage of large numbers is assured by selecting an appropriate magnification that will yield approximately 100 intercepts with the above-described 50 cm pattern. In addition, application of the central limit theorem permits the data to be interpreted as a normal distribution. The applicablility of normal statistics is substantiated by the fact that a chi-square test on the data showed that the measurement variation for the procedure is quite normal. Treating the measurements as a normal distribution provides a simple and relatively accurate means of qualifying the results. Thus, both accuracy and a confidence level are indicated for each grain size measurement.

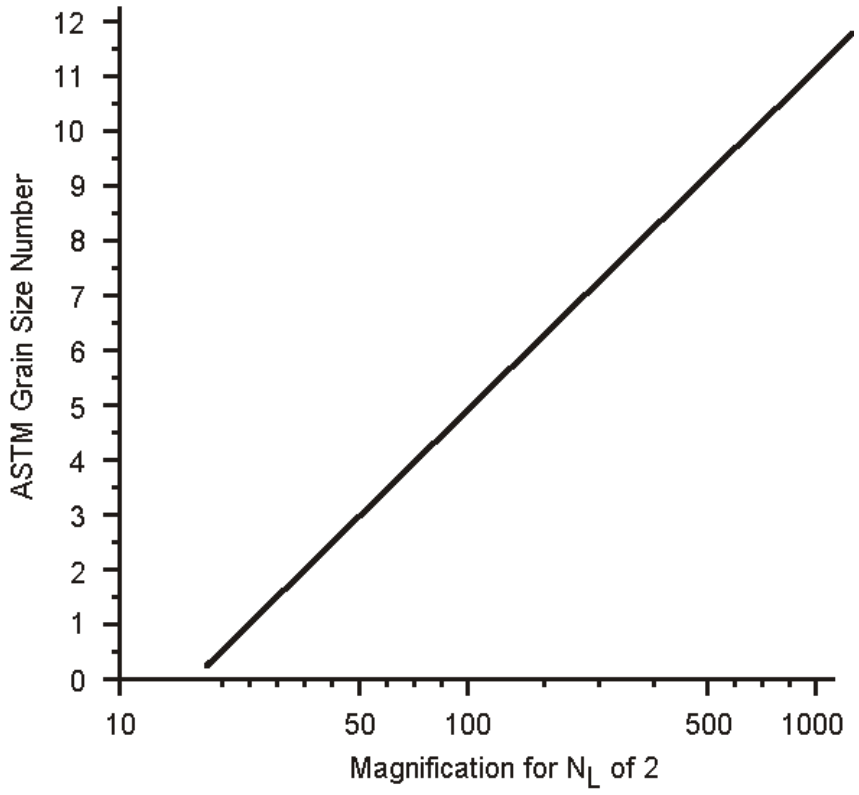

Figure 4
Relationship Between ASTM Grain Size Number & The Magnification To Provide Approximately 100 Intercepts On A 50 cm Pattern (NL of 2)

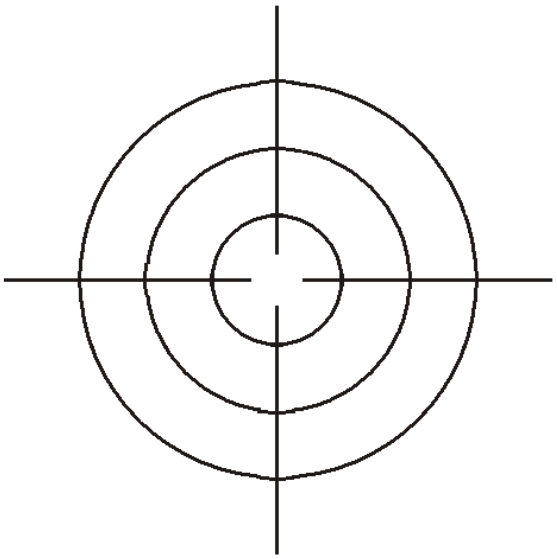

Figure 5
Circular Pattern With Total Length of 50 cm For Use With Intercept Method

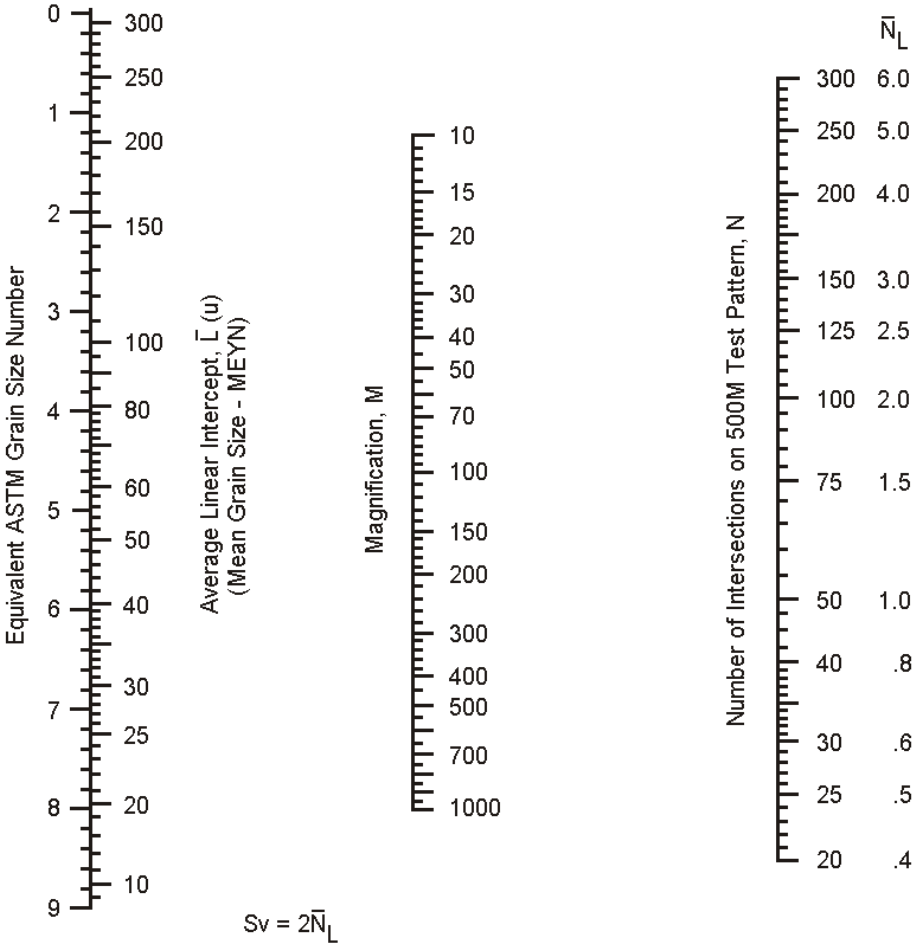

Figure 6
Nomograph To Convert Intercept Value To Grain Size

B. The step-by-step procedure for the method is outlined below, and amplification of each step is presented in the discussion segment.

1. Perform a cursory examination of the microstructure and estimate its equivalent grain size number using the comparison method, (Figures 12 and 13).

2. Refer to Figure 4 to ascertain the appropriate magnification that will yield approximately 100 intercepts for the 50-cm circular pattern shown in Figure 5. A nearby convenient magnification may be used.

3. Randomly select five areas on the specimen that will be used for counting to prevent biasing, the observer should not keep a consecutive count of the number of intercepts but should use a manual counter. Either direct measurement on the microscope using the appropriate reticule or measurement on the micrographs for permanent record may be used. Obtain five counts (X1 X5) of the grain size. Triple points should be given a double count; this will introduce a small systematic error, since 1.5 counts for triple points is technically correct. However, use of the manual counter, which does not permit half-counts, is considered important enough to justify this small error.

4. Calculate the average, the standard deviation, and the coefficient of variation using the following equations:

$$\bar{X} = \frac{\sum_{i=1}^{N} X_1}{N}$$

$$\sigma = \sqrt{\frac{(X_1 - \bar{X})^2}{N-1}}$$

$$C.V. = \frac{\sigma}{\bar{X}}$$

5. Convert the average number of intercepts, D, to the equivalent Heyn grain size in microns using Hilliard's nomograph adapted to our purposes Figure 6. For more accuracy, Figures 8 through 11, which are for the common magnification, may be used.

6. For the five fields observed (N=5), and the coefficient of variation calculated, use the curves shown in Figure 7 to qualify the measurement. Precise values for the accuracy and confidence level may be calculated by applying the equation which was used to generate the curves in Figure 7, namely:

$$N = \left(\frac{100\ Z_{c1}}{A} + \frac{\sigma x}{\bar{X}} \right)^2$$

Where: N = number of fields or number of independent measurements.
Z_{cl} = the number of standard deviations corresponding to a given confidence level for a normal distribution.
A = % accuracy or % deviation of estimated value from the "true" value.
σx = standard deviation of population.
$\bar{x}$ = Arithmetic mean of population.

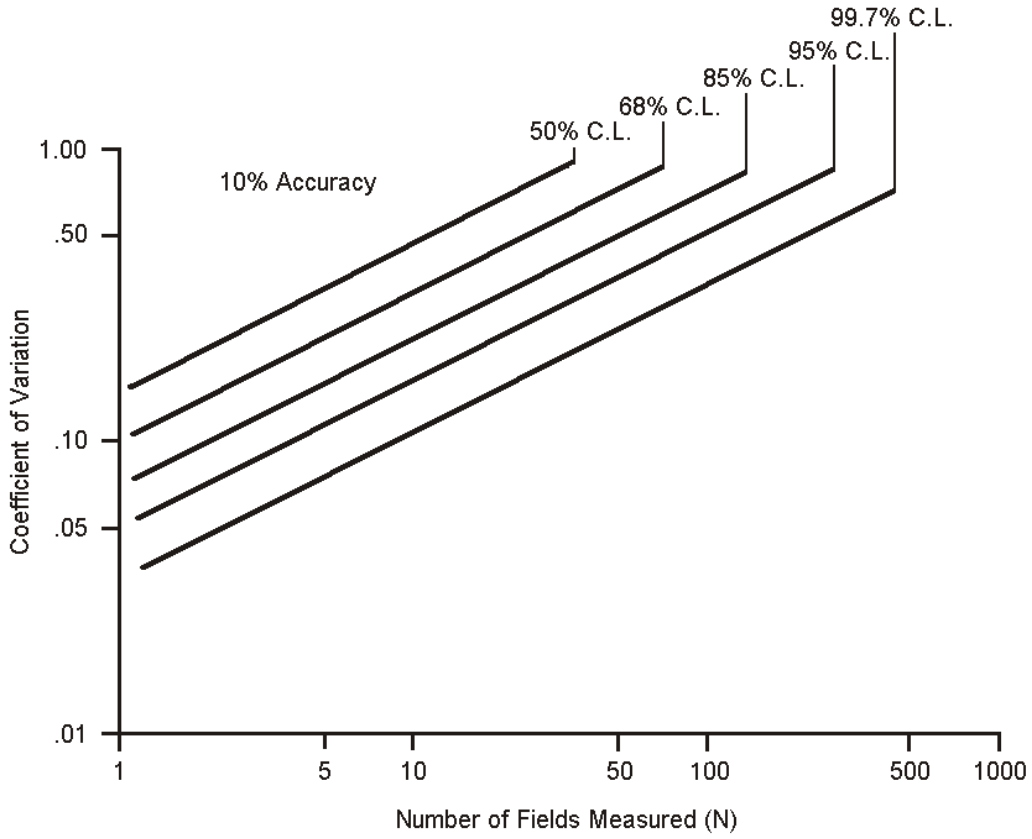

Figure 7
Coefficient Of Variation Versus The Number Of Fields Measured For A 5% Accuracy

7. If a desired accuracy and confidence level are not achieved by five measurements, then, since the coefficient of variation does not change very much as a function of N, Figure 7 can be used to determine approximately how many additional measurements are necessary.

Number Of Intercepts	0	1	2	3	4	5	6	7	8	9
50	100.0	98.0	96.2	94.3	92.6	90.9	89.3	87.7	86.2	84.7
60	83.3	82.0	80.6	79.4	78.1	76.9	75.8	74.6	73.5	72.5
70	71.4	70.4	69.4	68.5	67.6	66.7	65.8	64.9	64.1	63.3
80	62.5	61.7	61.0	60.2	59.5	58.8	58.1	57.5	56.8	56.2
90	55.6	54.9	54.3	53.8	53.2	52.6	52.1	51.5	51.0	50.5
100	50.0	49.5	49.0	48.5	48.1	47.6	47.2	46.7	46.3	45.9
110	45.5	45.0	44.6	44.2	43.9	43.5	43.1	42.7	42.4	42.0
120	41.7	41.3	41.0	40.7	40.3	40.0	39.7	39.4	39.1	38.8
130	38.5	38.2	37.9	37.6	37.3	37.0	36.8	36.5	36.2	36.0
140	35.7	35.5	35.2	35.0	34.7	34.5	34.2	34.0	33.8	33.6
150	33.3	33.1	32.9	32.7	32.5	32.3	32.1	31.8	31.6	31.4
160	31.3	31.3	30.9	30.7	30.5	30.3	30.1	29.9	29.8	29.6
170	29.4	29.2	29.1	28.9	28.7	28.6	28.4	28.2	28.1	27.9

Figure 8
Grain Size (Average Linear Intercept) in Microns for 50 cm Pattern at 100X

Number Of Intercepts	0	1	2	3	4	5	6	7	8	9
50	50.00	49.02	48.08	47.17	46.30	45.45	44.64	43.86	43.10	42.37
60	41.67	40.98	40.32	39.68	39.06	38.46	37.88	37.31	36.76	36.23
70	35.71	35.21	34.72	34.25	33.78	33.33	32.89	32.47	32.05	31.65
80	31.25	30.86	30.49	30.12	29.76	29.41	29.07	28.74	28.41	28.09
90	27.78	27.47	27.17	26.88	26.60	26.32	26.04	25.77	25.51	25.25
100	25.00	24.75	24.51	24.27	24.04	23.81	23.58	23.36	23.15	22.94
110	22.72	22.52	22.32	22.12	21.93	21.74	21.55	21.37	21.19	21.01
120	20.83	20.66	20.49	20.33	20.16	20.00	19.84	19.69	19.53	19.38
130	19.23	19.08	18.94	18.80	18.66	18.52	18.38	18.25	18.12	17.99
140	17.86	17.73	17.61	17.48	17.36	17.24	17.12	17.01	16.89	16.78
150	16.67	16.56	16.45	16.34	16.23	16.13	16.03	15.92	15.82	15.72
160	15.62	15.53	15.43	15.34	15.24	15.15	15.06	14.97	14.88	14.79
170	14.71	14.62	14.53	14.45	14.37	14.29	14.20	14.12	14.04	13.97

Figure 9
Grain Size (Average Linear Intercept) in Microns for 50 cm Pattern at 200X

Number Of Intercepts	0	1	2	3	4	5	6	7	8	9
50	25.00	24.51	24.04	23.58	23.15	22.73	22.32	21.93	21.55	21.19
60	20.83	20.49	20.16	19.84	19.53	19.23	18.94	18.66	18.38	18.12
70	17.86	17.61	17.36	17.12	16.89	16.67	16.45	16.23	16.03	15.82
80	15.62	15.43	15.24	15.06	14.88	14.71	14.53	14.37	14.20	14.04
90	13.89	13.74	13.59	13.44	13.30	13.16	13.02	12.89	12.76	12.63
100	12.50	12.38	12.25	12.14	12.02	11.90	11.79	11.68	11.57	11.47
110	11.36	11.36	11.26	11.16	11.06	10.96	10.78	10.68	10.59	10.50
120	10.42	10.33	10.25	10.16	10.08	10.00	9.92	9.84	9.77	9.69
130	9.62	9.54	9.47	9.40	9.33	9.26	9.19	9.12	9.06	8.99
140	8.93	8.87	8.80	8.74	8.68	8.62	8.56	8.50	8.45	8.39
150	8.33	8.28	8.22	8.17	8.12	8.06	8.01	7.96	7.91	7.86
160	7.81	7.76	7.72	7.67	7.62	7.58	7.53	7.49	7.44	7.40
170	7.35	7.31	7.27	7.23	7.18	7.14	7.10	7.06	7.02	6.98

Figure 10
Grain Size (Average Linear Intercept) in Microns for 50 cm Pattern at 400X

Number Of Intercepts	0	1	2	3	4	5	6	7	8	9
50	12.50	12.25	12.02	11.79	11.57	11.36	11.16	10.96	10.78	10.59
60	10.42	10.25	10.08	9.92	9.77	9.62	9.47	9.33	9.19	9.06
70	8.93	8.80	8.68	8.56	8.45	8.33	8.22	8.12	8.01	7.91
80	7.81	7.72	7.62	7.53	7.44	7.35	7.27	7.18	7.10	7.02
90	6.94	6.87	6.79	6.72	6.65	6.58	6.51	6.44	6.38	6.31
100	6.25	6.19	6.13	6.07	6.01	5.95	5.90	5.84	5.79	5.73
110	5.68	5.63	5.58	5.53	5.48	5.43	5.39	5.34	5.30	5.25
120	5.21	5.17	5.12	5.08	4.66	4.63	4.60	4.56	4.53	4.50
140	4.46	4.43	4.40	4.37	4.34	4.31	4.28	4.25	4.22	4.19
150	4.17	4.14	4.11	4.08	4.06	4.03	4.01	3.98	3.96	3.93
160	3.91	3.88	3.86	3.83	3.81	3.79	3.77	3.74	3.72	3.70
170	3.68	3.65	3.63	3.61	3.59	3.57	3.55	3.53	3.51	3.49

Figure 11
Grain Size (Average Linear Intercept) in Microns for 50 cm Pattern at 800X

5. DOCUMENTATION

5.1. Data sheet(s) shall contain the following:

- Title of test
- Specimen description including part number, lot number, etc.
- Test equipment used
- . Test procedure
- Values and observations
  - 109-89-1 Grain size estimate Figures 12 and 13
  - 109-89-2 Heyn's Intercept Method
    - Number of grains parallel to rolling direction
    - Number of grains perpendicular to rolling direction
    - Calculated average grain size
    - Photograph shall be included for material supplied to Material Specification 100-746
  - 109-89-3 Abram's Intercept Method
    - Number of intercepts per field
    - Average for 5 fields
    - Calculated average grain size
    - Photographs shall be included for material supplied to Material Specification 100-746
- Name of operator and date of test

5.2. The following shall be specified in the referencing document:

- Number of specimens to be tested shall be 1 specimen from each end of the coil received from supplier, except when material is supplied at die width; unless otherwise specified.
- Acceptance levels

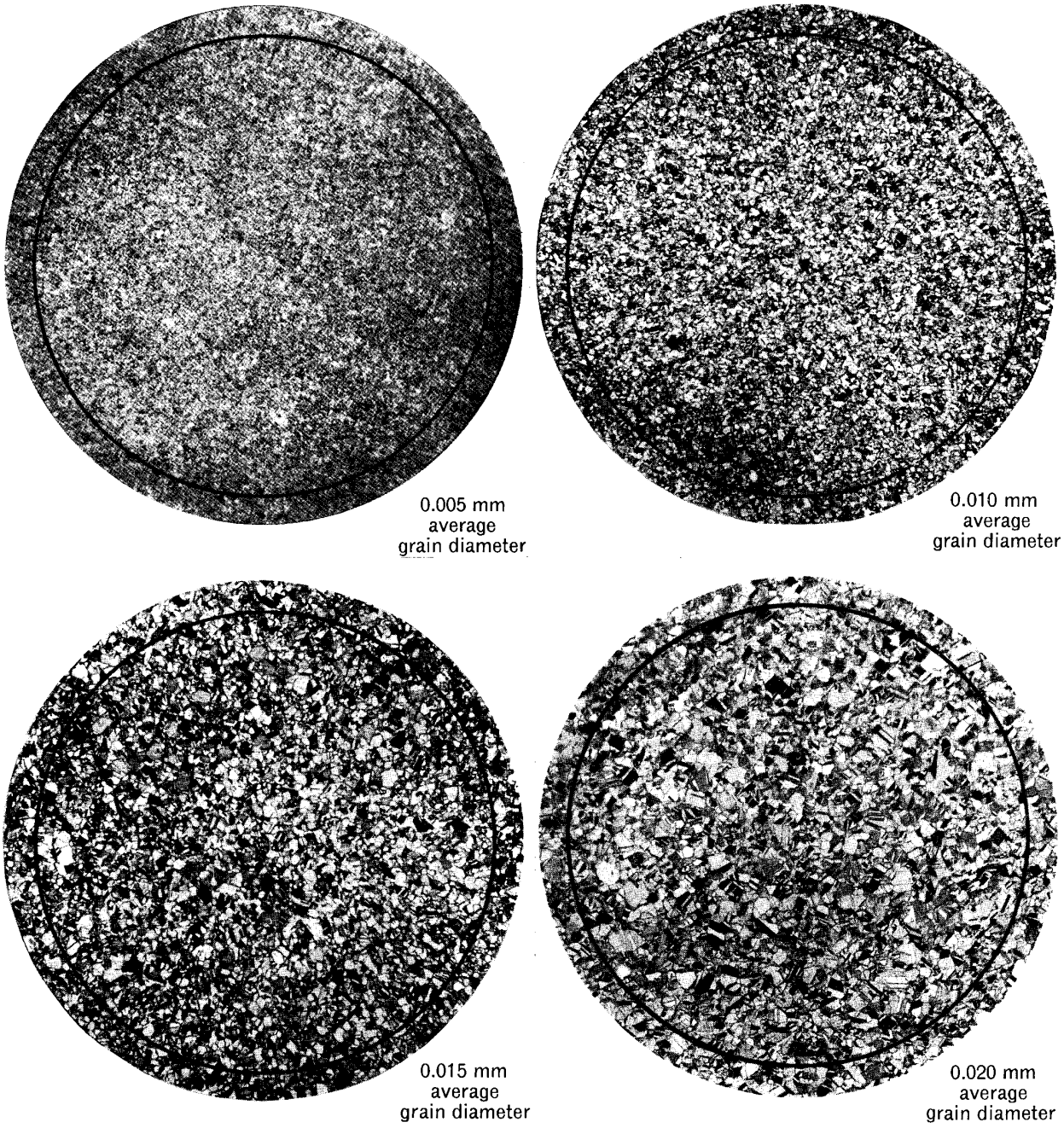

Figure 12 (continued)
Nonferrous, 75X

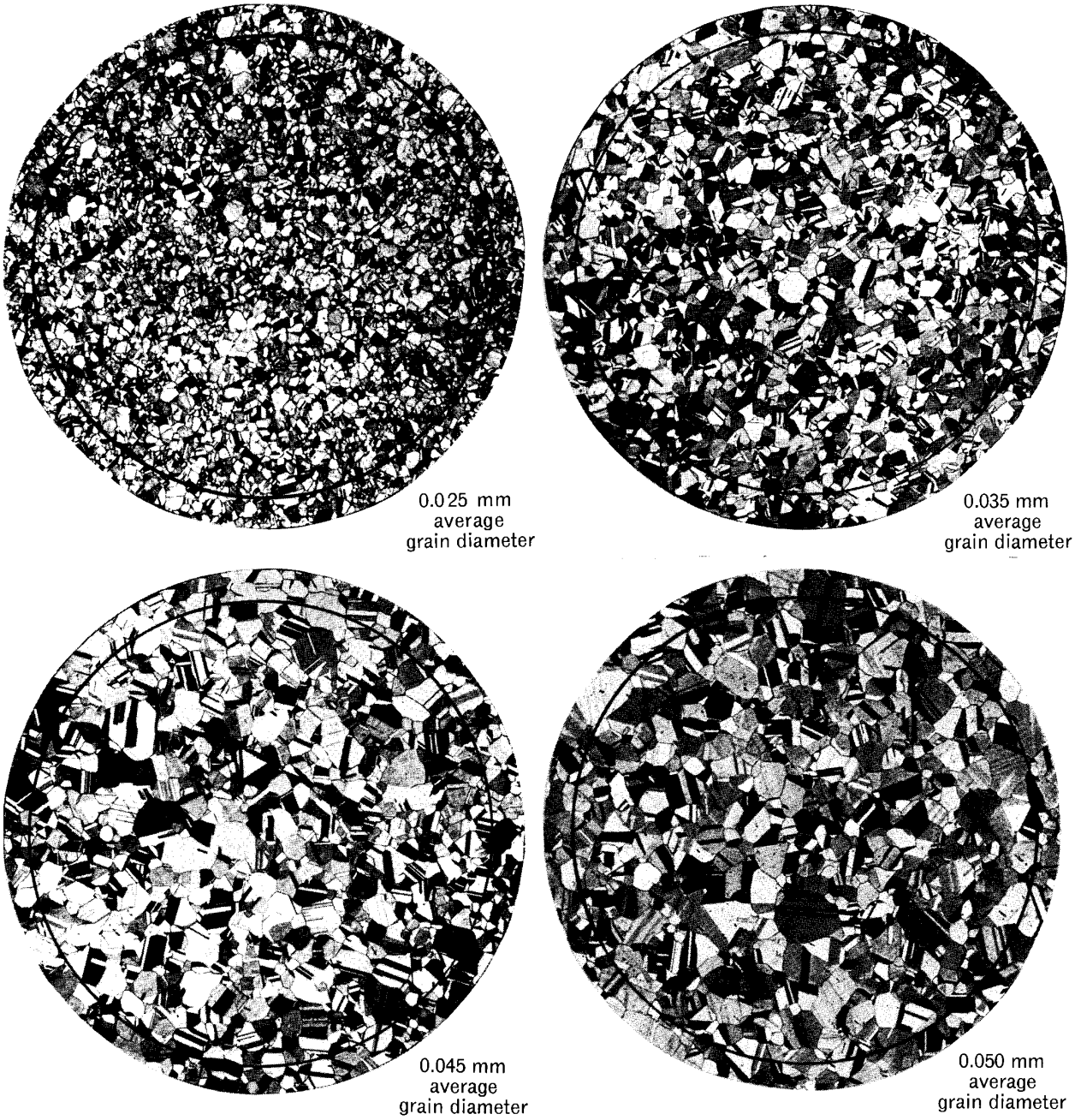

Figure 12 (continued)
Nonferrous, 75X

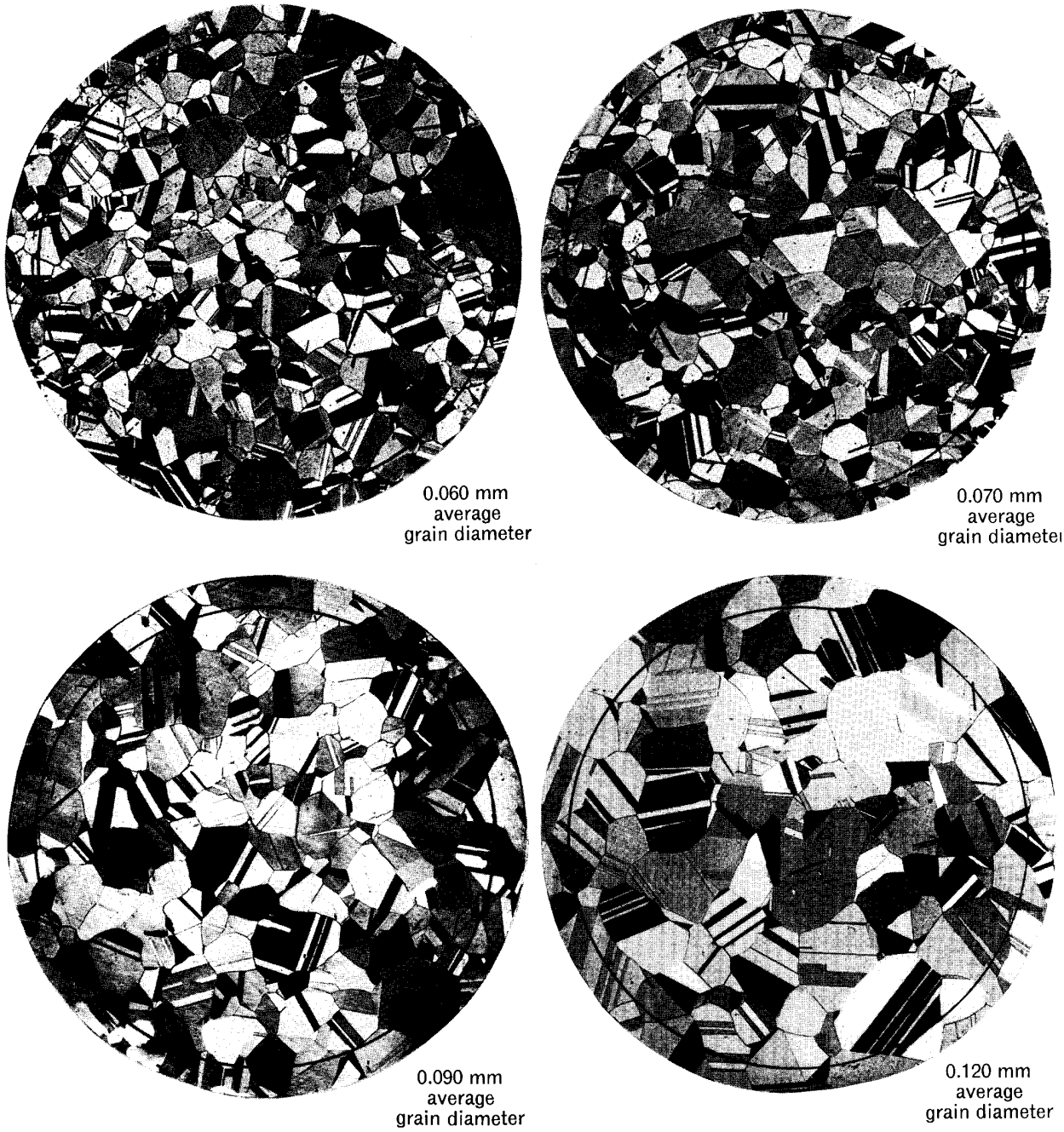

Figure 12 (end)
Nonferrous, 75X

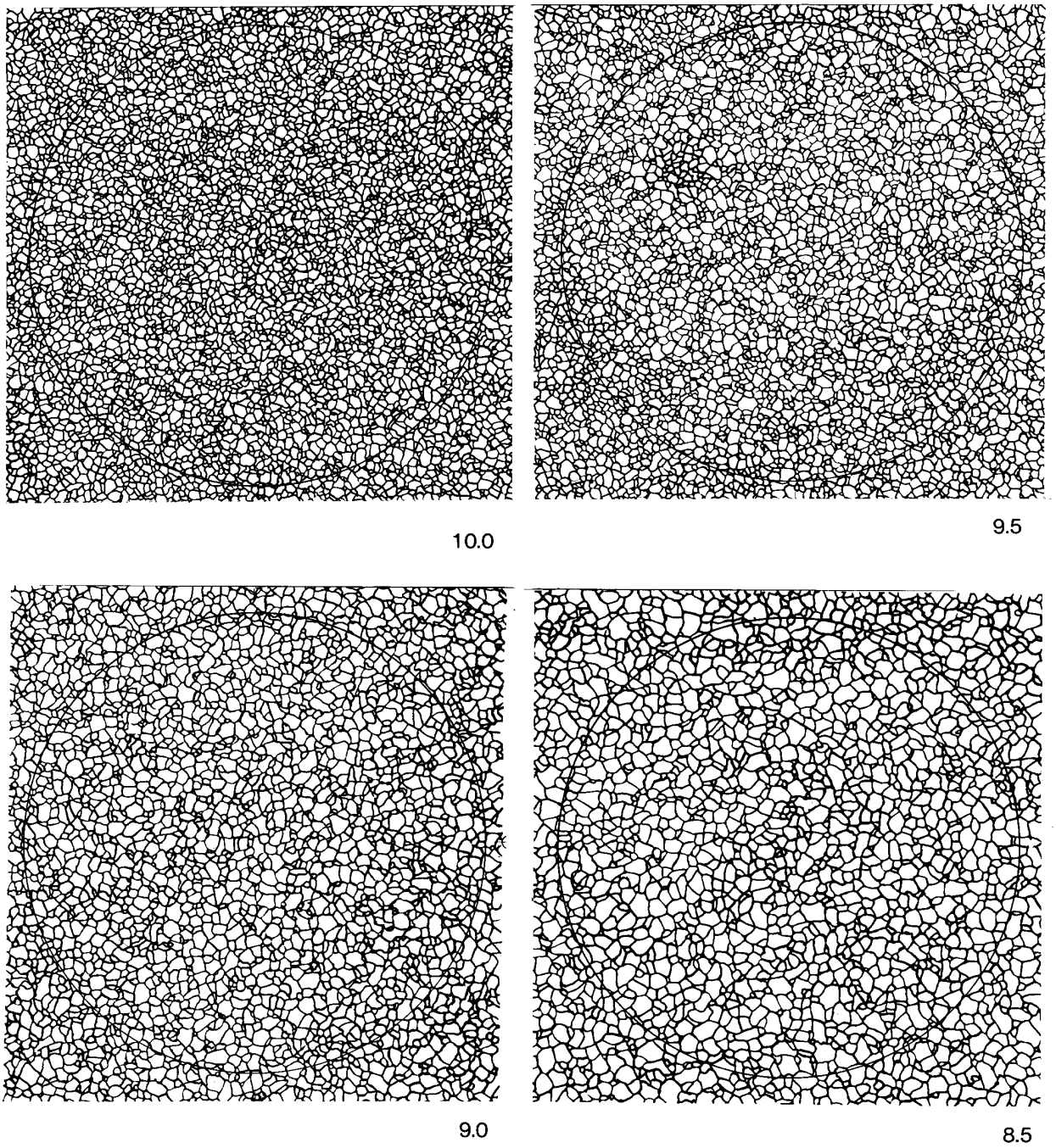

Figure 13 (continued)
Ferrous, 100X

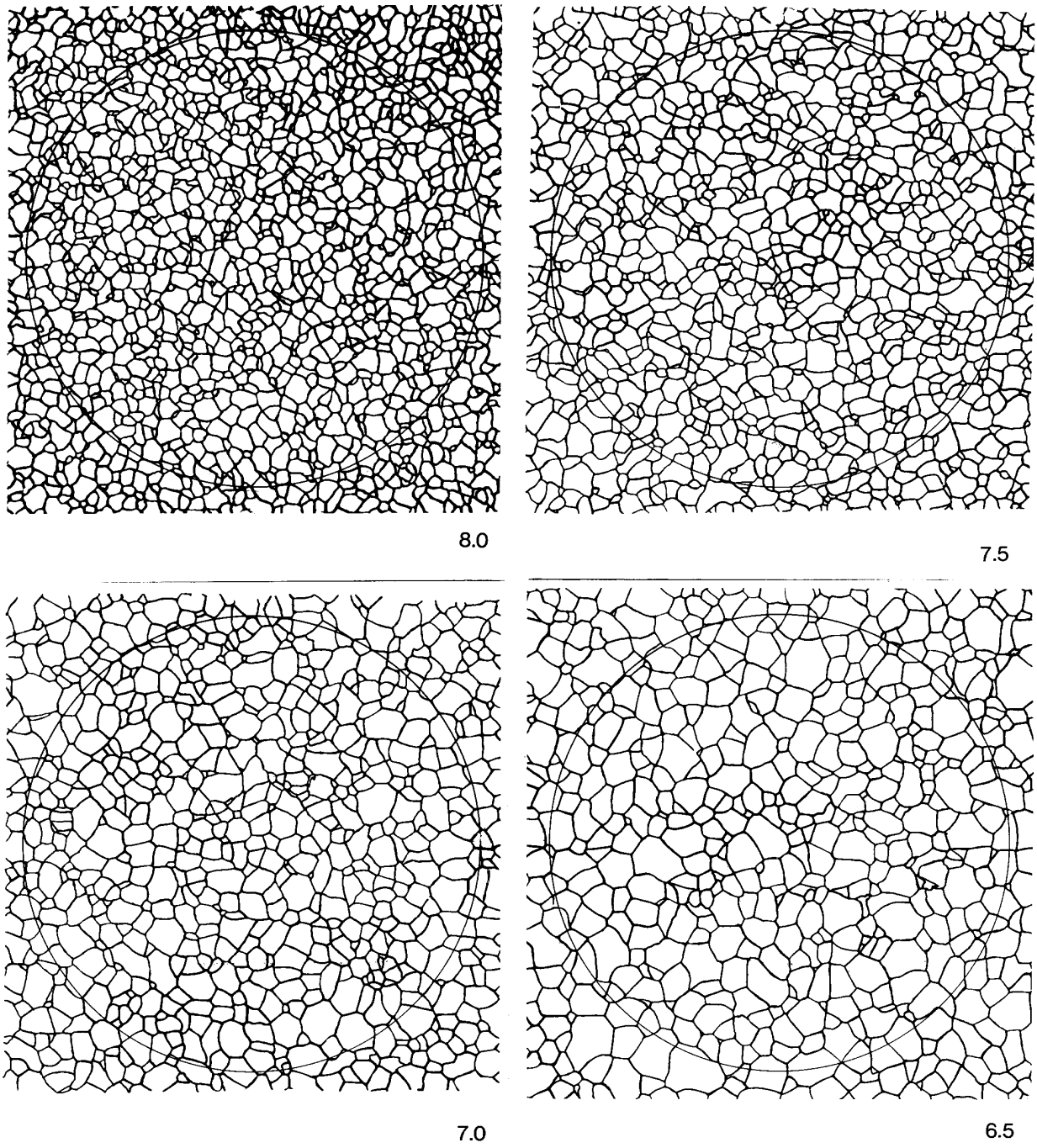

Figure 13 (continued)
Ferrous, 100X

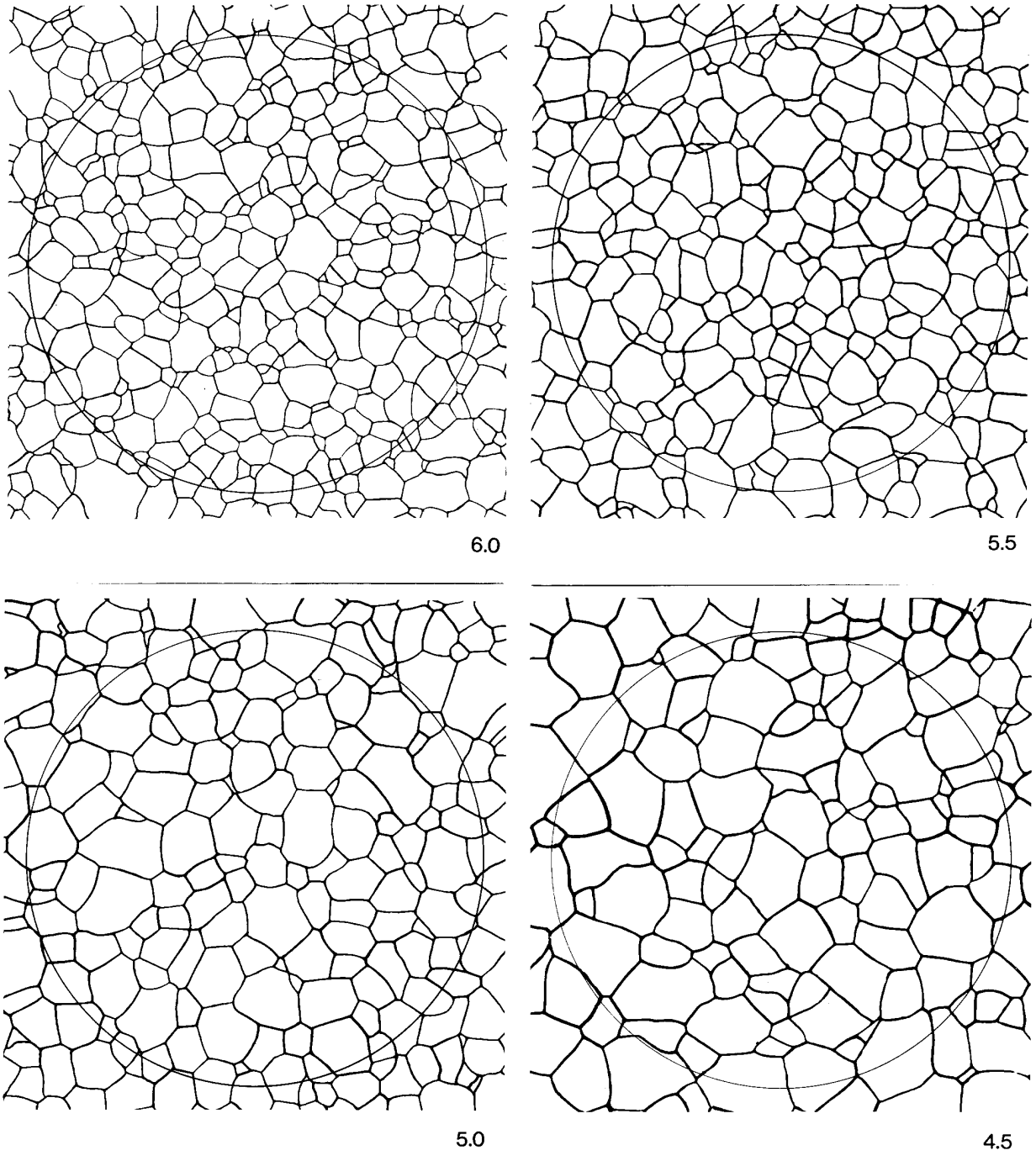

Figure 13 (end)
Ferrous, 100X